

Data Validation Checklist Inorganic Analyses

Project: 35TH Avenue Superfund Site
 Laboratory: TestAmerica - Savannah, GA
 Method: SW-846 6010C and 7471B; EPA 200.7 and 245.1
 Matrix: Soil and Water
 Reviewer: Jane Lindsey
 Concurrence¹: Carol Lovett

Project No: 15268508.20000
 Job ID.: 680-85980-2
 Associated Samples: Refer to Attachment A (Sample Summary)
 Date(s) Collected: 12/19/2012
 Date: 01/24/2013
 Date: 02/07/2013

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
1. Were sample preservation requirements met? If pH of aqueous sample >2 and was not adjusted by laboratory prior to analysis, J- flag positive results and R- flag non-detect results.	✓				
2. Were all COC records signed and integrity seals intact, indicating that COC was maintained for all samples?	✓				
3. Were there any problems noted in laboratory data package concerning condition of samples upon receipt?		✓			
4. Do any soil/sediment samples contain more than 50% water? If yes, then results are to be reported on a wet-weight basis.		✓			
5. Have any technical holding times, determined from date of collection to date of analysis, been exceeded? (Hg: ≤28 days, other metals: ≤6 months). If not, then J- flag positive results and R- flag non-detect aqueous results.		✓			
6. Were results for all project-specified target analytes reported?	✓				
7. Were project-specified Reporting Limits achieved for undiluted sample analyses?	✓				
8. Were method blank (MB) prepared at the appropriate frequency (one per 20 samples, batch, matrix, and level)?	✓				
9. Was a calibration blank (ICB/CCB) analyzed at the beginning, after every 10 th sample, and at the end of each analytical run?	✓				
10. Were target analytes detected in the method and/or calibration blanks?	✓			No analytes detected in the method blanks; calibration blanks were not evaluated.	
11. Were target analytes reported in equipment/rinsate blanks analyses above the DL?	✓			121912-RB-SIEVE (680-85980-46): Lead @ 150 µg/L (RL 10 µg/L, DL 4.0 µg/L); mercury @ 0.37 µg/L (RL 0.20 µg/L, DL 0.091 µg/L)	

¹ Independent technical reviewer

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
12. Were contaminants detected in samples below the blank contamination action level? <ul style="list-style-type: none"> ○ If blank result > RL, <ul style="list-style-type: none"> • Flag sample results \leq RL with a U • Flag positive sample results > RL and $\leq 10\times$ blank result, as J+ positive results ○ If blank result \leq RL, <ul style="list-style-type: none"> • Flag sample results \leq RL with a U • Flag positive sample results > RL and $\leq 10\times$ blank result, as J+ positive results 	✓			Refer to Attachment B (Sample-specific Blank Contamination Action Levels) Sample CV0627A-CS had a mercury result $\leq 10\times$ the RB result. Using professional judgment, the result was flagged with J rather than J+ because the MS/MSD recovery was below specified criteria.	J
13. Are there negative laboratory blank results with the absolute value \leq RL? If yes, then flag positive and non-detect sample results that are < 10x absolute blank value as J- and UJ, respectively.		✓			
14. Was a field duplicate analyzed?		✓			
15. Was precision deemed acceptable as defined by the project plans?			✓		
16. Were initial and continuing calibration standards analyzed at the lab/project-specified frequency for each instrument? <ul style="list-style-type: none"> ○ 6010C: <ul style="list-style-type: none"> • ICAL: Blank and one standard • ICV initially, and CCV every 10th sample and at the end of the analytical run • Lower Limit of Quantitation Check Sample (CRI) to be analyzed after establishing lower laboratory reporting limits and as needed ○ 7471B: <ul style="list-style-type: none"> • ICAL: Blank and five standards • ICV initially, and CCV every 10th sample and at the end of the analytical run 	✓				

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
<p>17. Were these results within lab/project specifications?</p> <ul style="list-style-type: none"> ○ 6010C <ul style="list-style-type: none"> • ICV/CCV (Criteria: 90-110%R): <ul style="list-style-type: none"> ▪ If %R <75, then J- flag positive results and R-flag non-detects ▪ If 75-89%R, then J- flag positive results and UJ flag non-detects ▪ If 111-125%R, then J flag positive results ▪ If >125%R, then J+ flag positive results ▪ If >160%R, then R flag positive results • CRI (Method: 70-130%R, Laboratory: 50-150%R; Project: 50-150%R for Sb, Pb, and Tl, and 70-130%R for all other analytes): <ul style="list-style-type: none"> ▪ If CRI %R <50 (<30% for Sb, Pb, TL), then R flag results $\leq 2x$ RL and J flag positive results $>2x$ RL ▪ If CRI %R 50-69% (30-49% for Sb, Pb, TL), then J- and UJ flag positive results $<2x$ RL and ND, respectively ▪ If CRI %R >130% and $\leq 180\%$ (>150%, but $\leq 200\%$ for Sb, Pb, TL), then J+ flag positive results $<2x$ RL ▪ If CRI %R >180% (>200% for Sb, Pb, TL), then R flag positive results ○ 7471B <ul style="list-style-type: none"> • ICV/CCV (Criteria: 80-120%R): <ul style="list-style-type: none"> ▪ If correlation coefficients <0.995, then J and UJ flag positive and non-detect results. ▪ If %R <65, then J- flag positive results and R-flag non-detects ▪ If 65-79%R, then J- flag positive results and UJ flag non-detects ▪ If 121-135%R, then J flag positive results ▪ If >135%R, then J+ flag positive results ▪ If >170%R, then R flag positive results • CRI (Method: Not required, Laboratory: 50-150%R, Project: 70-130%R): <ul style="list-style-type: none"> ▪ If CRI %R <50, then R flag results $\leq 2x$ RL and J flag positive results $>2x$ RL ▪ If CRI %R 50-69%, then J- and UJ flag positive results $<2x$ RL and ND, respectively ▪ If CRI %R >130% and $\leq 180\%$, then J+ flag positive results $<2x$ RL ▪ If CRI %R >180%, then R flag positive result 	✓				
18. Was the interference check sample (ICS) analyzed at the beginning of each ICP analytical run?	✓				

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
19. Are ICS recoveries within 80-120% of the true value? If not, qualify data as follows when native Al, Fe, Ca, and Mg sample concentrations are equal to or greater than the ICS spiking level: <ul style="list-style-type: none"> ○ If >120%R (or >true value plus 2x CRQL), J+ flag positive results ○ If 50-79%R (or less than true value – 2x the CRQL), J- flag positive results and UJ flag non-detects ○ If <50%R, J- flag positive results and R-flag non-detects 	✓				
20. Was a LCS analyzed for each preparation batch (one per 20 samples per matrix and level)?	✓				
21. Did LCS recoveries meet method/laboratory/project (80-120%R) specifications? <ul style="list-style-type: none"> ○ Soil: <ul style="list-style-type: none"> • LCS result > Upper control limit (UCL): J+ flag positive results • LCS result < Lower control limit (LCL): J- flag positive results and UJ flag non-detects ○ Aqueous: <ul style="list-style-type: none"> • If <50%R, then J- and R flag positive and ND results, respectively • If 50-LCL%R, J- and UJ flag positive and ND results, respectively • >UCL: J+ Flag positive results • >150%R: R Flag results 	✓				
22. Was the RPD between LCS and LCSD results within method/laboratory /project control limits ($\leq 20\%$ RPD)? If not, J and UJ flag positive and non-detect results, respectively			✓	LCS only	
23. Was a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) analyzed once per preparation batch?	✓				
24. Is the MS and MSD parent sample a project-specific sample?	✓	✓		<ul style="list-style-type: none"> • Solid, 6010C, Prep Batch 260659: FM0627A-CS (680-85980-2) • Solid, 7471B, Prep Batch 261384: FM0627A-CS (680-85980-2) • Aqueous, 200.7, Prep Batch 260769: 680-86038-1 (Batch sample) • Aqueous, 245.1, Prep Batch 261590: 680-85982-1 (Batch sample) 	

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
25. Was a post-digestion spike (PDS) analysis conducted when MS and/or MSD results did not meet control limits (Note: PDS is not required for silver)?	✓	✓		<ul style="list-style-type: none"> Solid, 6010C: FM0627A-CS (680-85980-2) Aqueous, 200.7: 680-86038-1 (Batch sample) 	
26. For all analytes with sample concentration < 4 x spike concentration, are spike recoveries within method (6010C: 75-125%R MS/MSD and 80-120%R PDS; 7471B: 80-120%R MS/MSD and PDS not required), laboratory (MS, MSD, and PDS: 75-125%R), and project (as noted below) specifications? <i>Only QC results for project samples are evaluated.</i> If not, <ul style="list-style-type: none"> 6010C: <ul style="list-style-type: none"> If MS %R <30 and PDS %R <75, then J- and R Flag positive and ND results, respectively If MS %R <30 and PDS %R >75, then J flag positive and UJ flag non-detect results If MS and MSD %R 30-74 and PDS%R <75, then J- flag positive and UJ flag non-detect results If MS and MSD %R 30-74 and PDS%R ≥75, then J flag positive and UJ flag non-detect results If MS, MSD, and PDS %R >125, J+ flag positive results If MS and MSD %R >125 and PDS %R ≤125, then J flag positive results If MS and MSD %R <30 and no PDS, then J- flag positive and R-flag non-detect results If MS and MSD %R 30-74 and no PDS, then J- and UJ flag positive and non-detect results, respectively If MS and MSD %R >125 and no PDS, then J+ flag positive results 7471B: <ul style="list-style-type: none"> If MS %R <30, then J- and R Flag positive and ND results, respectively If MS and MSD %R 30-74, then J- flag positive and UJ flag non-detect results If MS and MSD %R >125, then J+ flag positive results 		✓		CV0627A-CS (680-85980-39): <ul style="list-style-type: none"> Arsenic MSD @ -142%R (75-125). Qualification of data is not required, because MS %R (119) fell within control limits. Barium MS and MSD @ 173 and 244%R (75-125). The native sample concentration is greater than 4x the spiking level; therefore, an evaluation of interference is not possible. Chromium MSD @ 130%R. Qualification of data is not required, because MS %R (125) fell within control limits. Lead MS and MSD @ 60 and 723%R (75-125). The native sample concentration is greater than 4x the spiking level; therefore, an evaluation of interference is not possible. Mercury MS/MSD @ -70 and -14%R (80-120). Using professional judgment, the result was flagged with J, rather than J-, because mercury was detected within 10x the RB result. 	J
27. Were laboratory/project (≤20%RPD) criteria met for precision during the MS and MSD analysis? <i>Only QC results for project samples are evaluated.</i> <ul style="list-style-type: none"> If RPD >20%, J and UJ flag positive and non-detect results. 		✓		FM0627A-CS (680-85980-2): Mercury MS/MSD RPD @ 24% (≤20)	J
28. Was a serial dilution conducted for 6010C?	✓				

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
29. Is the serial dilution parent sample a project-specific sample?	✓	✓		<ul style="list-style-type: none"> • Solid, 6010C: 680-85929-6 (Batch sample). Lab sample 680-85929-6 is a project-specific sample (FM0266A-CS) that was selected by TestAmerica for serial dilution analysis, and the results were reported under Job ID 680-85929-1 • Aqueous, 200.7: 680-86038-1 (Batch sample) 	
30. Is the percent difference between the serially diluted result and undiluted result less 10% (for those analytes with native concentrations greater than 50x the DL)? <i>Only QC results for project samples are evaluated.</i> <ul style="list-style-type: none"> ○ If %D >10, J and UJ flag positive and non-detect results, respectively. 	✓				
31. Was a laboratory duplicate analyzed?		✓			
32. Was the lab duplicate analysis conducted on a project-specific sample?			✓		
33. Were criteria for laboratory/project precision met? <i>Only QC results for project samples are evaluated.</i> <ul style="list-style-type: none"> ○ If RPD values >20% (35% for soil/sediment) or absolute difference > RL (2x RL for soil/sediment), then J and UJ flag positive and non-detect results, respectively 			✓		
34. Were lab comments included in report? If yes, summarize contents or attach a copy of the narrative.	✓			Refer to Attachment C (Case Narrative)	
Comments: The data validation was conducted in accordance with the <i>Non-Industrial Use Property Sampling Event QAPP for the 35th Avenue Removal Site, Birmingham, Alabama, Revision 1</i> (OTIE, October 2012). The data review process was modeled after the <i>USEPA Contract Laboratory Program (CLP) National Functional Guidelines (NFG) for Inorganic Data Review</i> (EPA 540-R-04-004, October 2004). Sample results have been qualified based on the results of the data review process (Attachment D). Criteria for acceptability of data were based upon available site information, analytical method requirements, guidance documents, and professional judgment					

DV Flag Definitions:

J-	The result is an estimated quantity, but the result may be biased low.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
U	The analyte was analyzed for, but was not detected above the associated level; blank contamination may exist.
UJ	The analyte was analyzed for, but was not detected. The reported limit is approximate and may be inaccurate or imprecise.

ATTACHMENT A
SAMPLE SUMMARY

Sample Summary

Client: Oneida Total Integrated Enterprises LLC
Project/Site: 35th Avenue Superfund Site

TestAmerica Job ID: 680-85980-2
SDG: 68085980-2

Lab Sample ID	Client Sample ID	Matrix	Collected	Received
680-85980-39	CV0627A-CS	Solid	12/19/12 09:15	12/20/12 10:27
680-85980-46	121912-RB-SIEVE	Water	12/19/12 12:00	12/20/12 10:27

ATTACHMENT B

SAMPLE-SPECIFIC BLANK CONTAMINATION ACTION LEVELS

Sample-Specific Blank Contamination Action Levels

Attachment B

6010C:

6010C:				CV0627A-CS	
				85980-39	
				%S:	72.2
				DF:	1
				Wt, g:	1.04
Analyte	RL, µg/L	Blank Results, µg/L	Maximum Amount Detected ¹ , µg/L	Vol , ml:	100
		121912-RB- Sieve		Action Level ² , µg/L	Sample-Specific Blank Contamination Action Levels, mg/Kg
Lead (Pb)	10	150	150	1500	200
Reported Sample Result:					250
Reporting Limit, mg/Kg:					1.3
Blank Contamination Action:					None

Rinsate blank results reported in micrograms per liter (µg/L). Laboratory blank results and sample-specific blank contamination action levels reported in milligrams per

¹ Maximum amount detected in among all blanks

² Maximum amount detected in blanks multiplied by a factor of 10

Blank Contamination Actions:

- o If blank result > RL,
 - Flag sample results ≤ RL with a U
 - Flag positive sample results > RL and ≤10x blank result , as J+ positive results
- o If blank result ≤RL,
 - Flag sample results ≤ RL with a U
 - Flag positive sample results > RL and ≤10x blank result , as J+ positive results

7471B:

7471B:				CV0627A-CS	
				85980-39	
				%S:	72.2
				DF:	1
				Wt, g:	0.58
Analyte	RL, µg/L	Blank Results, µg/L	Maximum Amount Detected ¹ , µg/L	Vol , ml:	50
		121912-RB-Sieve		Action Level ² , µg/L	Sample-Specific Blank Contamination Action Levels, mg/Kg
Mercury	0.2	0.37	0.37	3.7	0.4418
Reported Sample Result:					0.35
Reporting Limit, mg/Kg:					0.024
Blank Contamination Action:					J+

ATTACHMENT C
CASE NARRATIVE

Case Narrative

Client: Oneida Total Integrated Enterprises LLC
Project/Site: 35th Avenue Superfund Site

TestAmerica Job ID: 680-85980-2
SDG: 68085980-2

Job ID: 680-85980-2

Laboratory: TestAmerica Savannah

Narrative

CASE NARRATIVE

Client: Oneida Total Integrated Enterprises LLC
Project: 35th Avenue Superfund Site
Report Number: 680-85980-2

With the exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. In addition all laboratory quality control samples were within established control limits, with any exceptions noted below. Each sample was analyzed to achieve the lowest possible reporting limit within the constraints of the method. In some cases, due to interference or analytes present at high concentrations, samples were diluted. For diluted samples, the reporting limits are adjusted relative to the dilution required.

Calculations are performed before rounding to avoid round-off errors in calculated results.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

RECEIPT

The samples were received on 12/20/2012; the samples arrived in good condition, properly preserved and on ice. The temperature of the coolers at receipt was 4.2 C.

METALS (ICP)

Sample 121912-RB-SIEVE (680-85980-46) was analyzed for Metals (ICP) in accordance with EPA Method 200.7.

TOTAL MERCURY

Sample 121912-RB-SIEVE (680-85980-46) was analyzed for total mercury in accordance with EPA Method 245.1.

Method(s) 245.1: The method blank associated with batch 680-261590 contained mercury greater than one-half the reporting limit (RL). The data have been qualified and reported.

METALS (ICP)

Sample CV0627A-CS (680-85980-39) was analyzed for Metals (ICP) in accordance with EPA SW-846 Method 6010C.

Method(s) 6010C: The matrix spike / matrix spike duplicate (MS/MSD) recoveries for batch 680-260659 were outside control limits for arsenic and chromium. The associated laboratory control sample (LCS) recovery met acceptance criteria.

Method(s) 6010C: Due to the high concentration of barium and lead, the matrix spike / matrix spike duplicate (MS/MSD) for batch 680-260659 could not be evaluated for accuracy and precision. The associated laboratory control sample (LCS) met acceptance criteria.

TOTAL MERCURY

Sample CV0627A-CS (680-85980-39) was analyzed for total mercury in accordance with EPA SW-846 Method 7471B.

Method(s) 7471A, 7471B: The matrix spike / matrix spike duplicate (MS/MSD) recoveries for batch 261384 were outside control limits. The associated laboratory control sample (LCS) recovery met acceptance criteria.

Method(s) 7471B: The matrix spike / matrix spike duplicate (MS/MSD) percent recoveries and %RPD for batch 680-261384 were outside control limits for mercury.

ATTACHMENT D

QUALIFIED SAMPLE RESULTS

Client Sample Results

Client: Oneida Total Integrated Enterprises LLC
Project/Site: 35th Avenue Superfund Site

TestAmerica Job ID: 680-85980-2
SDG: 68085980-2

Client Sample ID: CV0627A-CS

Lab Sample ID: 680-85980-39

Date Collected: 12/19/12 09:15

Matrix: Solid

Date Received: 12/20/12 10:27

Percent Solids: 72.2

Method: 6010C - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Arsenic	36		2.7	0.79	mg/Kg	☼	12/21/12 13:03	12/26/12 16:37	1
Barium	240		1.3	0.40	mg/Kg	☼	12/21/12 13:03	12/26/12 16:37	1
Cadmium	1.9		0.67	0.13	mg/Kg	☼	12/21/12 13:03	12/26/12 16:37	1
Chromium	37		1.3	0.67	mg/Kg	☼	12/21/12 13:03	12/26/12 16:37	1
Lead	250		1.3	0.71	mg/Kg	☼	12/21/12 13:03	12/26/12 16:37	1
Selenium	2.4	J	3.3	1.3	mg/Kg	☼	12/21/12 13:03	12/26/12 16:37	1
Silver	1.3	U	1.3	0.13	mg/Kg	☼	12/21/12 13:03	12/26/12 16:37	1

Method: 7471B - Mercury in Solid or Semisolid Waste (Manual Cold Vapor Technique)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Mercury	0.35	J	0.024	0.0098	mg/Kg	☼	12/28/12 18:54	12/31/12 11:59	1

Client Sample ID: 121912-RB-SIEVE

Lab Sample ID: 680-85980-46

Date Collected: 12/19/12 12:00

Matrix: Water

Date Received: 12/20/12 10:27

Method: 200.7 Rev 4.4 - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Arsenic	20	U	20	4.6	ug/L	—	12/22/12 09:26	12/26/12 23:55	1
Barium	10	U	10	2.3	ug/L	—	12/22/12 09:26	12/26/12 23:55	1
Cadmium	5.0	U	5.0	2.0	ug/L	—	12/22/12 09:26	12/26/12 23:55	1
Chromium	10	U	10	1.2	ug/L	—	12/22/12 09:26	12/26/12 23:55	1
Lead	150		10	4.0	ug/L	—	12/22/12 09:26	12/26/12 23:55	1
Selenium	20	U	20	6.4	ug/L	—	12/22/12 09:26	12/26/12 23:55	1
Silver	10	U	10	0.89	ug/L	—	12/22/12 09:26	12/26/12 23:55	1

Method: 245.1 - Mercury (CVAA)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Mercury	0.37	B	0.20	0.091	ug/L	—	01/02/13 10:50	01/04/13 21:32	1

Sample results have been qualified by URS in accordance with the Non-Industrial Use Property Sampling Event QAPP for the 35th Avenue Removal Site, Birmingham, Alabama, Revision 1 (OTIE, October 2012)